

**MISSOURI DEPARTMENT OF NATURAL RESOURCES  
DIVISION OF ENVIRONMENTAL QUALITY  
ENVIRONMENTAL SERVICES PROGRAM  
Standard Operating Procedures**

SOP #: MDNR-FSS-230                      EFFECTIVE DATE: July 1, 2004

SOP TITLE: Field Operation of the HazMatID Infrared Spectroscopy Instrument

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SUMMARY OF REVISIONS: Not applicable - this is a new SOP.

APPLICABILITY:                      The procedures described in this SOP are applicable to all  
   ESP personnel who need to analyze samples using the  
   HazMatID infrared spectroscopy instrument.

DISTRIBUTION:                      MoDNR Intranet  
   ESP SOP Coordinator

RECERTIFICATION RECORD:

Date Reviewed				
Initials				

## 1.0 SCOPE AND APPLICABILITY

The procedures outlined in this document are applicable to operation of the HazMatID infrared spectroscopy instrument by ESP personnel. The HazMatID is a field screening Fourier Transform Infrared (FT-IR) spectrometer that, when used with other field screening techniques, may allow ESP personnel to qualitatively identify unknown chemical compounds. Liquid or solid samples may be identified by this method.

Several HazMatID instruments were purchased with Homeland Security funding through grants administered by the State Emergency Management Agency. The intended purpose of obtaining the equipment is for ESP Environmental Emergency Response staff to provide support to the Missouri Homeland Security Teams at potential Homeland Security related incidents.

## 2.0 SUMMARY OF METHOD

Unknown samples must initially be screened using field instruments such as pH meters, photoionization detectors, combustible gas indicators, and radiation meters in an effort to classify or characterize the unknown. If these measurements do not conclusively classify or characterize the unknown, the HazMatID can be used as another tool to attempt to identify the unknown. The HazMatID instrument passes an infrared beam from a laser through a diamond crystal and through the sample, then reflects the beam back to the detector. Covalent chemical bonds absorb infrared energy at characteristic wavelengths for the particular atoms associated with the bond. This results in a spectrum of the unknown material that maps the types of covalent bonds present in the unknown. The spectrum of the unknown is compared to known compounds contained in libraries on the HazMatID. The system provides up to ten potential identifications of the unknown, ranking each in order of descending correlation. A perfect correlation is 1.00. Correlation results of 0.95 or greater are considered to be potential positive identifications. Correlation values from 0.85 to 0.95 are indicative of the class of compound, and not necessarily a positive identification. Correlation's of less than 0.85 are not reliable and must not be used as a positive identification of the unknown.

## 3.0 INTERFERENCES AND LIMITATIONS

The HazMatID is capable of identifying many organic and inorganic compounds in both solid and liquid form. It does however have the following interferences and limitations:

- 3.1 The HazMatID is intended to provide initial determinations and should be used as an information resource in the field and not for absolute or conclusive identifications of unknown substances. The results provided by the HazMatID should be verified by using other appropriate techniques.
- 3.2 Samples containing significant water will not be easily identified because water obscures critical areas of the spectrum used for positive identification of other chemicals.

- 3.3 The HazMatID cannot identify elemental substances such as iron or mercury.
- 3.4 The HazMatID cannot identify ionic salts such as sodium chloride or calcium chloride.
- 3.5 The HazMatID cannot positively identify biological agents because of the complex nature of their composition. However, the instrument does provide a warning that indicates when biological material (protein) may be present in a sample.
- 3.6 The HazMatID generally cannot identify individual components of any mixture. While the instrument allows the subtraction of one component of a mixture, the residual spectrum may not be able to be identified.
- 3.7 Any potential positive identifications are limited to compounds that are contained in the HazMatID libraries. It cannot identify compounds that are not contained in the libraries.

#### 4.0 DEFINITIONS AND ACRONYMS

- CFR – Code of Federal Regulations
- EER – Environmental Emergency Response Section
- EPA – Environmental Protection Agency
- ESP – Environmental Services Program
- FT-IR – Fourier Transform Infrared
- HASP – Health and Safety Plan
- HAZWOPER – Hazardous Waste Operations Emergency Response
- HSERP – Hazardous Substance Emergency Response Plan
- MoDNR – Missouri Department of Natural Resources
- NIOSH – National Institute for Occupational Safety and Health
- OSHA – Occupational Safety and Health Administration
- PID – Photoionization Detector
- PPE – Personal Protective Equipment
- SOP – Standard Operating Procedure

#### 5.0 HEALTH AND SAFETY WARNINGS

Due to the nature of the environment in which the HazMatID may be placed during operation, extreme caution should be taken with respect to respiratory and explosion hazards. The following precautions should always be taken:

- 5.1 The chemicals to be identified by the HazMatID may be highly flammable or toxic. The instrument operator must always use appropriate personal protective equipment (PPE). A site specific Health and Safety Plan (HASP) must be prepared for sites where hazardous wastes or substances may be encountered and must describe the

level of protection needed for site workers. If the chemical hazards are unknown, then a high level of protection is needed (level B or higher). If field screening results or other knowledge identifies a lower risk, then the level of PPE may be downgraded. At a minimum, protective gloves and safety glasses should be worn when operating the instrument.

5.2 The HazMatID is not rated intrinsically safe for explosive environments. Therefore, the HazMatID must not be used in environments where a risk of explosion has been identified.

5.3 The HazMatID must be decontaminated after every use. Refer to the operator's manual for details.

## 6.0 PERSONNEL QUALIFICATIONS

All ESP personnel directly involved in field investigations at sites that fall under the EPA Worker Protection requirements of 40 CFR part 311, referencing OSHA 29 CFR Part 1910.120, and meet the definition of HAZWOPER activities must:

- Attend a 40-hour course designed to meet OSHA health and safety training requirements for hazardous waste site workers;
- Attend an annual 8-hour health and safety refresher course, or receive equivalent training;
- Participate in the ESP medical monitoring program;
- Receive appropriate on-the-job training, and pass a proficiency test before using the HazMatID alone;
- Be familiar with the HSERP, written and maintained by the ESP; and
- Be familiar with the SOP manual and have read all SOP documents that are applicable to the field activities, including but not limited to those referenced in this SOP.

## 7.0 EQUIPMENT AND SUPPLIES

- PPE gear
- pH paper
- PID meter
- Combustible gas meter
- Radiation detector
- HazMatID system including charged batteries
- Isopropyl Alcohol swabs
- Acetone
- Polypropylene Test Film
- Spatula
- Liquid well
- Volatiles cover
- Laptop Computer (optional)

## 8.0 PROCEDURE

- 8.1 The HazMatID must always be stored and transported in the black Pelican carrying case provided with the instrument. The only time the instrument should be out of the case is when it is in use.
- 8.2 Batteries for the HazMatID should not be stored in the unit when it is not in use. Storing the batteries in the unit will slowly discharge them even though the system is not turned on. Store the batteries in the charger provided for them so that they are fully charged and ready for use. Each battery will have approximately two hours of use when fully charged, and require 8 to 12 hours to re-charge.
- 8.3 The compartment containing the batteries on the HazMatID must be sealed when the unit is being operated. Failure to seal the compartment can result in chemical contamination of the battery compartment, which is difficult to decontaminate.
- 8.4 Refer to the manufacturer's operations manual for specific instrument operations.

## 9.0 OPERATION OF THE HAZMATID

- 9.1 Remove the HazMatID from the black Pelican carrying case, install a charged battery, and seal the battery compartment.
- 9.2 Turn the power switch on. The system will take approximately 10 minutes to boot up.
- 9.3 The HazMatID instrument has an on-board computer. There is also a laptop computer for remote operation via a wireless connection. It is acceptable to link the laptop and on-board computers any time data is being collected, but that is not the preferred mode of operation. In most field situations, all data will be collected using the on-board computer. If the data requires any further manipulation, the data should be transferred from the on-board computer to the laptop computer for processing. It should be noted that all software functions can be performed on either the laptop or the on-board computer.
- 9.4 The laptop computer should be used if the operator is in a level of PPE that restricts hand movement to a degree that hinders operation of the HazMatID from the on-board computer. In this situation, two operators are required. The laptop computer should be linked to the on-board computer. The first operator will be in appropriate PPE in the exclusion zone placing samples on the HazMatID. The second operator will be in the support zone with the laptop actually using the software to analyze the samples.
- 9.5 If the laptop computer is to be used, install the wireless network card in the laptop and turn on the computer. When the computer has booted up to the desktop, double click on the icon **Shortcut to Connect**. Once the connection is established, the

laptop computer and the on-board computer will have the same display on their screens. At this point the HazMatID can be operated from either computer, and each computer will track all HazMatID operations.

- 9.6 A screen appears that prompts the entry of a username and password. Usernames and passwords are assigned to each operator of the system after successful completion of their proficiency testing.
- 9.7 Press **Keyboard** next to username and type in the assigned username, and then press **Done**.
- 9.8 Press **Keyboard** next to the password and type in the assigned password, and then press **Done**.
- 9.9 Check that the username and password are correct and if so press **Login**. A home screen will appear with the options of **Start**, **Advanced Features**, and **Logout**. There will also be two boxes in the upper left-hand corner labeled *Status* and *Battery*. The *Status* box should be green and indicate ready, and the *Battery* should show the estimated battery life in minutes. If the *Status* box is yellow or red, refer to the troubleshooting section of this SOP for corrective action.
- 9.10 Select **Advanced Features**, and then select **Method Manager**. A drop-down menu appears with the methods stored on the HazMatID. All methods on the system will be created prior to proficiency testing for each operator. Operators should never have to create or edit a method on their own. A fact sheet listing the description and application of each method will be provided with each unit. Highlight the method to be used, press **Select Method**, then press **Done**. Select **Home** to return to the screen to begin sample analysis.
- 9.11 To begin sample analysis, select **Start**. A screen will appear prompting the operator to clean the sample area and sensor. Use an isopropyl alcohol swab to clean the areas, then select **Continue**.
- 9.12 The HazMatID will begin collecting background spectrum and the user is prompted to enter Incident (the EER incident #), Sample ID (use consecutive numbers starting with 01), and Comments (site name, location). Using the **Keyboard** function in the same manner as it was used for username and password, type in the requested information, then select **Continue**.
- 9.13 If the sample to be analyzed is liquid, place several drops of the unknown directly onto the sample area, enough to cover the diamond. The liquid well or volatiles cover may have to be utilized if the unknown appears to evaporate quickly. Pressing **Continue** will collect the sample spectrum of the unknown.
- 9.14 When solid samples are to be analyzed, place a small amount of the unknown onto the sample area, enough to cover the diamond, and lower the pressure device down

on the unknown until it locks into place. Press **View Camera** to make sure the unknown covers the entire sampling area. Pressing **Continue** will collect the sample spectrum of the unknown.

- 9.15 When the sample analysis is complete, a result screen will appear that lists up to ten possible identifications of the unknown. The identifications are ranked by correlation from highest to lowest. Correlations of 0.95 to 1.00 are potential positive identifications. Correlations of 0.85 to 0.95 are indicative of a class of compound but not a positive identification. The operator must always confirm that the HazMatID identification is accurate or definitive by comparing the result(s) from the HazMatID to the initial screening test results and observations about the sample.
- 9.16 The HazMatID cannot positively identify biological agents because of the complex nature of their composition. The system does provide a warning that indicates biological material may be present by being very sensitive to proteins. If a sample containing protein is placed on the HazMatID, the following warning will appear on the screen: ***Warning! The Spectrum has features that are consistent with a protein and may contain biological material. (Protein check).*** This warning will appear in red print at the bottom of the result page. The operator must assume at this point that the material may be a biological hazard and take appropriate precautions.
- 9.17 It is possible to visually compare the unknown spectra to one or more of the ten possible identifications. From the result screen, select **Visual Compare**. A screen will appear that shows the unknown spectra and the highlighted library spectra on the same screen. The operator can scroll down the library spectra one at a time and compare each one to the unknown spectra. Press **Done** to return to the result screen.
- 9.18 If the unknown is believed to be a mixture of two different compounds, the operator has the option of subtracting one identified library entry from the unknown spectra to try and identify one of the two compounds present. From the result screen, select the library entry to be subtracted from the unknown and press **Search Residual**. A second screen appears with the library entry and the unknown. Press **Search Residual** and the software will provide the best hit for the residual spectrum. To return to the result screen, press **Cancel**.
- 9.19 It is possible to get NIOSH information about the identified compounds. To do this the operator needs to highlight one of the library entries on the result page and select **NIOSH Information**. If the compound is in the NIOSH library, a screen will appear listing the pertinent information. Select **Done** to return back to the result page
- 9.20 To run additional samples, select **Home** to return to the home page. Return to section 9.11 of this SOP and follow the sequence from section 9.11 to section 9.15

for analysis of additional unknown samples.

- 9.21 After all samples have been analyzed, the HazMatID should be powered down. After the system is powered down, the battery should be removed. The system is then placed back in the black Pelican carrying case for transport and storage. ***It is very important to power down the system in the proper sequence to avoid corrupting the software on the system. Complete power-down instructions are in section 9.22.***
- 9.22 To initiate power down from the result page, select **Home** which will return the system to the home page. From the home page, select **Logout**, which will take the system to the login page. From the login page, select **Shutdown**, which returns the system to a page where the operator is given two options. If **Home** is selected, the system returns to the page where sample analysis can begin. If **Shutdown** is selected, the power down sequence continues, and the system goes to the Windows desktop. From the **Start** icon in the lower left corner of the Windows desktop, select **Turn off Computer**. Select **Turn off** from the pop-up box that appears. All windows applications will end, and after two minutes a message will appear indicating it is safe to turn off the computer. It is safe at this point to turn off the computer with the power switch, remove the battery, and place the instrument in the carrying case.

## 10.0 ADDING ENTRIES TO USER LIBRARIES

- 10.1 It is possible to add entries to user created libraries on the HazMatID system. Creating library entries will be done by designated system administrators within the ESP.
- 10.2 When the operator of a HazMatID collects a spectra that they feel should be added to the user library, they should place a copy of the spectra in the following location on the ESP computer network: P:\EER\HazMatID Data\Region\County\Incident Number.
- 10.3 The operator should contact an ESP system administrator via e-mail and inform the administrator of the location of the sample spectra.
- 10.4 No compound will be added to any user library until such time as its identification has been confirmed by the technical support group of the manufacturer, or by a confirmatory chemical analysis by a laboratory
- 10.5 System administrators will periodically update all HazMatID libraries on all systems within the ESP so that each system will be identical.



## 11.0 TROUBLESHOOTING

- 11.1 The HazMatID is designed to require little in the way of maintenance by the operator. The most important issue is to thoroughly clean the sample area with isopropyl wipes between samples to prevent cross contamination.
- 11.2 The most common problem will result from a gradual misalignment of the mirrors used to direct the infrared beam during sample analysis. Misalignment results in a gradual weakening of the signal that will compromise sample identification. The operator can run a diagnostic that will re-align the mirrors. This should only be performed when the system performance and the intensity count indicate it is needed (i.e., when the Energy count drops below 20,000).
- 11.3 Every HazMatID is shipped from the factory with an intensity count of 26,500. This can be checked from the home page by selecting **Advanced Features**, then selecting **Diagnostics**. A screen will appear that has four boxes on the left side labeled Energy, Battery, Source, and Laser. For proper operation, all of the boxes should be green. The Energy box will have the actual energy displayed in it, and will be green so long as the energy count is above 20,000. The Battery box should be in countdown mode indicating estimated battery life in minutes. The Source and Laser boxes should both have the word pass in them.
- 11.4 If the energy intensity is below 20,000, the box color will change from green to yellow. The system will actually run so long as the energy count is above 10,000. If the count drops below 10,000, the box will turn red and the system will not run.
- 11.5 If it is decided to try to increase the energy count above 20,000, select **Automatic Alignment**. The system will run through a diagnostic that will hopefully increase energy intensity above 20,000.
- 11.6 The Source and Laser boxes are simply pass or fail, and the operator can do nothing to fix them. If they are in fail mode, a second diagnostic can be run from the same page. This diagnostic will not fix the problem, but will provide information to provide to the manufacturer so they can assess the extent of the problem. This diagnostic will only be performed with assistance from a system administrator.
- 11.7 When diagnostics are complete, select **Done**, then select **Home** to return to the home page.
- 11.8 If these diagnostic procedures fail to remove any error messages, or increase the energy intensity to a level at which the system will operate properly, the system will have to be sent back to the manufacturer for service.

## 12.0 QUALITY ASSURANCE AND QUALITY CONTROL

The HazMatID is a qualitative system and not quantitative so no calibration actually exists for the system. Analyzing known compounds can assess that the instrument is operating properly. Liquid system performance can be verified by using acetone and demonstrating that it properly identifies the compound. Solid sample performance can be demonstrated by analyzing a polypropylene film provided with the system. Analysis of known materials (i.e., acetone, polypropylene) should be completed prior to each use of the instrument with the results recorded in the user's field notebook. If the system fails to verify known compounds, then the operator should refer to section 11 for troubleshooting.

## 13.0 TECHNICAL SUPPORT

The manufacturer of the HazMatID maintains a technical assistance program called REACHBACK. This program provides a 24-hour hotline that exists to answer any questions anyone may have concerning the HazMatID or the data generated from it. This service requires a subscription, which ESP currently has. There are two telephone numbers to call to access the REACHBACK program. The first number is (866) 442-0628. The second number is (866) 777-8880. Actual spectra can be sent to REACHBACK for interpretation by e-mailing them to the following address: [reachback@sensir.com](mailto:reachback@sensir.com).

## 14.0 REFERENCES

- Appropriate MDNR Field Services Section Standard Operating Procedures for field screening techniques.
- HazMatID System User's Guide
- HazMatID Embedded Application Software
- QualID Application Software User's Guide
- HSERP